VI.

CONTRIBUTIONS FROM THE CHEMICAL LABORATORY OF HARVARD COLLEGE.

ON THE VAPOR-DENSITY OF THE CHLORIDE, THE BROMIDE, AND THE IODIDE OF ANTIMONY.

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The usual form of the tube employed in Victor Meyer's method for determining vapor-densities was found unsatisfactory, in that any slight variation in the tension or the temperature of the enclosed air — such as was caused by drawing out the stopper or dropping in the substance — was apt to draw water up the delivery arm from the pneumatic trough, and so crack the hot tube. This was remedied by enlarging the delivery arm both in length and in bore. Several modifications of the tube were tried, varying in capacity from 75 to 200 cubic centimeters. The tube that was found most satisfactory was of hard glass, of the usual form (with the exception just noted), and of about 150 c.c. capacity.

The neck was closed with a perforated rubber stopper, into which was fitted a thin glass tube drawn out and closed at one end, the other end being thrust nearly through the bored stopper. The substance was weighed out in a small tube of the same size as that just described, and the open mouth of this weighing tube was thrust into the lower end of the same perforation; so that the rubber stopper both held and closed it. The stopper being tightly fitted into the neck, and the apparatus heated to a constant temperature, the upper tube was forced downwards, pushing before it the tube holding the substance, which was thus dropped into the bulb of the apparatus. As soon as the displaced air ceased to come over, the closed tip of the upper tube was broken off, thus effectually preventing the back flow from the pneumatic trough on cooling the apparatus or drawing the stopper.

Bromide of Antimony, $SbBr_3$.

The substance used being a white crystalline solid, not deliquescent and not readily oxidized, no special precautions were found necessary.

Results.

No. 1	12.71
No. 2	12.34
No. 3	12.66
Mean	12.57
Theory	12.48
Greatest difference	0.14
Mean difference	0.09

Chloride of Antimony, $SbCl_3$.

Since the chloride of antimony is very deliquescent, the substance was weighed in a closed tube, which was afterwards quickly transferred to the perforated stopper of the apparatus, arranged as above described so as effectually to protect the material from the atmosphere, till the tube was dropped into the heated bulb. Determinations were made in both air and nitrogen; but the results seemed to show that no advantage was gained by using nitrogen.

Results.

No. 1	8.06
No. 2	8.03
No. 3	7.80
Mean	7.96
Theory	7.85
Greatest difference	0.16
Mean difference	0.11

Iodide of Antimony, SbI_3.

Pure material was prepared by subliming in a stream of carbonic dioxide a quantity of an otherwise pure substance, which, by exposure to air and light, had been partly changed into the oxyiodide. Several

determinations were made in the air, but the results showed that, at the temperature of volatilization, the iodide was rapidly oxidized, and the determinations were afterwards made in an atmosphere of nitrogen. Many unsuccessful attempts were made to obtain pure nitrogen by the well-known method described by Dr. Wolcott Gibbs. The method consists in simply mixing together and warming solutions of sodium nitrite, ammonium nitrate (or sulphate) in excess, and potassium dichromate in large excess, the speed of the reaction being regulated by the amount of the dichromate added. It was found that red fumes always appeared in the nitrogen evolved; and that, while no variations of proportions quite eliminated the fumes, still, when but very little bichromate was used, the resulting nitrogen was comparatively pure. It was evident, as Dr. Gibbs pointed out, that the nitric fumes were caused by chlorine present as an impurity in the materials used, and the impurity was readily traced to the dichromate; but, as the purification of potassium dichromate is troublesome and expensive, it was found more practicable to pass the impure nitrogen over red-hot copper filings recently reduced, and then through drying-tubes. The nitrogen thus purified gave satisfactory results.

Results.

No. 1.	17.25
No. 2.	17.90
No. 3.	17.63
Mean.	17.59
Theory	17.33
Greatest difference . . .	0.31
Mean difference	0.26

It will be seen that the results do not agree very closely with the theory, or with each other. While we do not presume that we have obtained as accurate results as the method will allow, a few trials carefully conducted and closely watched made it evident that very close results cannot be reasonably expected from this method. An absolutely constant temperature — indicated by the unchanging position of the water surface in the delivery tube — it is practically impossible to maintain. Furthermore, the slight resistance to be overcome by the air in rushing out of the delivery tube is often enough to retain

the last bubbles, especially when there is any delay in the volatilization of the substance, giving time for the diffusion and partial condensation of the vapor. Such variable conditions seem to preclude the possibility of uniformly accurate and consistent results from Meyer's method.